भारतीय मानक
Indian Standard

IS 101 (Part 8/Sec 4) : 2015
(Reaffirmed 2020)

रोगनों वार्निशों और सम्बद्ध उत्पादों के नमूने लेने और परीक्षण की पद्धतियाँ

भाग 8 वर्णकों और अन्य ठोसों के परीक्षण

अनुभाग 4 थैलिक एनहाईड्राईड

(चौथा पुनरीक्षण)

Methods of Sampling and Test for Paints, Varnishes and Related Products

Part 8 Tests for Pigments and Other Solids

Section 4 Phthalic Anhydride

(Fourth Revision)

ICS No. 87.040; 87.060.10

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December 2015 **Price Group 2**

Paints, Varnishes and Related Products Sectional Committee, CHD 20

FOREWORD

This Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

This standard is one of series dealing with methods of sampling and testing of paints, varnishes and related products.

In this revision, method of separation of resin by centrifuge method is provided which was not present in the 1993 version of the test method. In earlier version there was confusion about the mass of the sample taken for the test, that is whether it is of the total paint sample taken for the test or of the non-volatile vehicle. In this version mass of the resin has been taken into consideration for test. This has been done to remove any confusion about identity of the material being weighed. Non-volatile of the resin solution has been included in the final calculation since phthalic anhydride percent by mass is calculated on non-volatile basis.

The Committee responsible for formulation of this standard is given in Annex B.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance, with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 4 Phthalic Anhydride Content

(Fourth Revision)

1 SCOPE

This standard (Part 8/Sec 4) prescribes the method to determine the phthalic anhydride content in the paint.

2 REFERENCES

The standards listed in Annex A contains provisions which though reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 APPARATUS

3.1 Centrifuge Tube — 50 ml, heavy walled.

3.2 Water Bath — Electrically operated and thermostatically controlled.

3.3 Air Oven — Electrically operated and thermostatically controlled shall be capable of being maintained at a temperature 105 ± 2°C.

3.4 Analytical Balance — of suitable range having least count 0.1 mg.

3.5 Centrifugal Machine — Capable of swirl at minimum 5 000 rpm.

3.6 Flask and Condenser — A 500 ml long-necked flask fitted with water condenser. The joint between the flask and the condenser shall be a taper ground glass joint.

3.7 Desiccator — With concentrated sulphuric acid as desiccant.

3.8 Evaporating Disc — of 500 ml capacity.

3.9 G4 Glass Crucibles (*see* IS 5011)

3.10 Soda Lime Guard Tube

4 REAGENTS

4.1 Benzene (*see* IS 534)

4.2 Methyl Alcohol (*see* IS 517)

4.3 Acetone (*see* IS 170)

4.4 Petroleum Ether (*see* IS 1745)

4.5 Toluene — LR Grade

4.6 Alcoholic Potassium Hydroxide Solution — Dissolve 66 g of potassium hydroxide in 1 000 ml of absolute alcohol.

4.7 Ether — Anhydrous, LR Grade (*see* IS 336)

4.8 Alcohol–Toluene Wash Solution — 1:3 (*v*/*v*).

4.9 Hydrochloric Acid — 0.1 N (*see* IS 265)

4.10 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

5 PROCEDURE

5.1 Weigh accurately 15 to 20 g of the well mixed material into a weighed centrifuge tube. Centrifuge the mixture as per IS 101 (Part 8/Sec 2) to isolate the resin part. Collect the resin part in evaporating disc and concentrate, add some boiling chips during this process.

5.2 Calculate the non-volatile content of the resin as per IS 101 (Part 8/Sec 2).

5.3 Weigh the resin solution (M_2) sufficient to yield 0.8 to 1.2 g of alcoholic potassium phthalate solution into a 500 ml long-necked flask. Add 150 ml of benzene, warm if necessary, over a water bath and bring into solution. Add 60 ml of alcoholic potassium hydroxide solution. Reflux for 1 h over a water bath using the condenser. Remove the flask from the water bath, rinse down the inside of the condenser with a few ml of alcohol-toluene wash solution. Remove the condenser and stopper of the flask with soda lime guard tube and cool the flask to below 20°C. Filter the contents though a weighed sintered G4 Glass Crucible, when cool. Use alcohol-toluene wash solution to transfer the precipitate completely from the flask to crucible. Wash the precipitate with successive portions

of alcohol-toluene wash solution until a few mililitre of wash solution shows no sign of alkalinity to Phenolphthalein solution. Do not allow to draw air though the crystals as they are hygroscopic.

5.4 Finally wash the precipitate with 25 ml of ether. Wipe the outside of the crucible with a clean cloth and place in an oven at 60°C for 1 h. The precipitate is alcoholate and alcohol of crystallization may be driven off on prolonged heating. However, it is safe to dry up to 60°C for 1 h. Cool to room temperature in a desiccators, weigh (M_1) and calculate as follows:

$$\text{Phthalic anhydride contents by mass} = \frac{(M_1 \times 51.36 \times 100)}{(M_2 \times \text{percent NVM of resin solution})}$$

where

M_1 = of the precipitate obtained, mass in g; and

M_2 = of the resin taken for test, mass in g.

NOTE — *Correction for carbonate*: Co-precipitation of potassium carbonate (K_2CO_3) with potassium alcohol phthalate may be source of error. If a correction of K_2CO_3 is desired then dissolve precipitate of potassium phthalate alcoholate in 50 ml distilled water that has been neutralized to phenolphthalein indicator solution and if solution is alkaline , titrate with 0.1 N HCL. Calculate correction factor K as,

K = (Volume of HCl × normality of HCl) × 0.138 2

$$\text{Phthalic anhydride content by mass} = \frac{(M_1 - K) \times 51.36 \times 100}{(M_2 \times \text{percent NVM of resin solution})}$$

5.4.1 The result is represented as the percentage of phthalic anhydride in solid resin obtained from the paint.

5.5 Alternately heat the precipitate of alcoholate at 150°C for 2 h. All alcohol molecules will be driven off, weigh the precipitate (M_1) which is $C_6H_4(COOK)_2$ and phthalic anhydride percent may be calculated as follows:

$$\text{Phthalic anhydride present by mass} = \frac{(M_1 \times 61.16 \times 100)}{(M_2 \times \text{percent NVM of resin solution})}$$

where

M_1 = mass in g of the precipitate obtained.

M_2 = mass in g of the resin taken for test.

5.5.1 The result is represented as the percentage of phthalic anhydride present in solid resin obtained from the paint.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 101 (Part 8/ Sec 2) : 1990	Method of sampling and test for paints, varnishes and related products: Part 8 Tests for pigments and other solids, Section 2 Pigments and non-volatile matter
IS 170 : 2004	Acetone — Specification (*fourth revision*)
IS 265 : 1993	Hydrochloric acid (*fourth revision*)
IS 336 : 1973	Specification for ether (*second revision*)
IS 517 : 1986	Specification for methanol(methyl alcohal) (*second revision*)
IS 534 : 2007	Benzene — Specification (*fourth revision*)
IS 1070 : 1992	Reagent grade water (*third revision*)
IS 1745 : 1978	Specification for petroleum hydro-carbon solvent (*second revision*)
IS 5011 : 1968	Gooch crucibles

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

Paints, Varnishes and Related Products Sectional Committee, CHD 20

Organization	*Representative(s)*
National Test House, Kolkata	Dr Sunil Kumar Saha (*Chairman*)
Akzo Nobel Coatings India Pvt Ltd, Bengaluru	Dr T. Kanakaraju
Asian Paints Ltd, Mumbai	Dr B. P. Mallik
Berger Paints India Ltd, Howrah	Dr B. Bera
Bharat Heavy Electricals Ltd, Tiruchirapalli	Shri L. Gragori
Central Building Research Institute, New Delhi	Dr S. P. Agarwal Dr P. C. Thapliyal (*Alternate*)
Central Public Works Department, New Delhi,	Shri Vijay Motwanihri S. P. Chaudhary (*Alternate*)
Clariant Chemicals (India) Ltd, Thane	Shri Nitin Vaidya Shri Umesh Kapoor (*Alternate*)
Consumer Association of India, Chennai	Shri N. Gopalaswami Shri R. Desikan *(Alternate*)
Consumer Unity and Turst Society (CUTS), Jaipur	Ms Keya Ghosh Shri Vijay Singh *(Alternate*)
Directorate of Naval Architecture, New Delhi	Commodore R. Ghosh Commander R. K Sehgal (*Alternate*)
Engineers India Limited, New Delhi	Shri R. Chaudhury Shri A. Satya Shridhar (*Alternate*)
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Indian Institute of Technology, Mumbai	Dr A. S. Khanna
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Kerala Minerals and Metals Ltd, Kollam Kerala	Shri Rajendra Prasad Dr John George (*Alternate*)
Maruti Udyog Ltd, Gurgaon	Shri S. S. Nirmaan
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Ministry of Defence, Department of Standardization, New Delhi	Shri S. K. Kaibarta Shri V. K. Chhabra (*Alternate*)
Ministry of Industry, DIPP, New Delhi	Shri N. C. Tiwari
National Test House (ER), Kolkata	Shrimati Ratna De Sarkar
Naval Materials Research Laboratory (NMRL), Thane	Shri Dhirendra Kumar Shri V. R. More (*Alternate*)
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Paint and Coating Technologists Association, Kanpur	Shri V. N. Dubey
Punjab Paint Colour and Varnish Works Pvt Ltd, Kanpur	Shri G. N. Tiwari
Research Designs & Standards Organization, Lucknow	Shri C. Sengupta Shri R. K. Bhagchandani (*Alternate*)

Organization	*Representative(s)*
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Member Secretary
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Scientist 'E' (CHD), BIS

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 20 (1805).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi